SEGMENTATION OF THE OUTER CONTACT ON P-TYPE COAXIAL GERMANIUM DETECTORS

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ABSTRACT

Germanium detector arrays are needed for low-level counting facilities. The applications of such user facilities include characterization of low-level radioactive samples. In addition, the same detector arrays can perform important fundamental physics measurements including the search for rare-events like neutrinoless double-beta decay. Germanium coaxial detectors having segmented outer contacts can provide sensitivity improvement in low-background measurements. The segmented outer detector contact allows pulse-shape analysis measurements that provide additional background reduction. Currently, n-type (reverse electrode) germanium coaxial detectors are used whenever a segmented coaxial detector is needed because the outer boron (electron barrier) contact is thin and can be relatively easily segmented. Coaxial detectors fabricated from p-type germanium cost less, usually have better energy resolution, and can be larger than n-type coaxial detectors. However, it is difficult to reliably segment p-type coaxial detectors because thick (~ 1 mm) lithium-diffused (hole barrier) contacts are the standard outside contact for p-type coaxial detectors. During this Phase II small business innovation research grant (SBIR), we are developing thinner, segmented, and stable lithium-diffused contacts. Many small planar test detectors have been fabricated with segmented lithium-diffused contacts. Center contact and guard-ring structures have been fabricated on the detectors to study the rectification and segmentation properties of lithium-diffused contacts. Fabrication techniques have been established that successfully produce repeatable rectification and segmentation. The rectification and segmentation appear to be stable with respect to modest temperature cycles. These results are fundamental steps toward developing segmented lithium-diffused contacts for p-type coaxial detectors. Large segmented p-type coaxial detectors based on this technology could serve as gamma-ray spectrometers on instruments such as the Radionuclide Aerosol Sampler / Analyzer (RASA). These detectors will provide more sensitive, lower background, measurements than currently available unsegmented p-type coaxial detectors.

OBJECTIVES

Germanium detector arrays are needed for low-level counting facilities. The practical applications of such user facilities include characterization of low-level radioactive samples. In addition, the same detector arrays can also perform important fundamental physics measurements including the search for rare events like neutrino-less double-beta decay (Miley et al., 1991; Miley, et al., 1990; Majorana Collaboration White Paper, 2003; and Goulding et al., 1984). Coaxial germanium detectors having segmented outer contacts could provide the next level of sensitivity improvement in low background measurements. The segmented outer contact allows performance of advanced pulse-shape analysis measurements. These techniques can be used to discriminate between multiple Compton-scattered gamma-ray events and single-point beta-decay events. Because of their complexity, segmented coaxial detectors are expensive and available only after relatively long lead times. Improved detector segmentation techniques would be both important and timely. Such technological advances will reduce fabrication costs and improve availability of these detectors for the low-level counting community.

Currently, n-type (reverse electrode) germanium coaxial detectors are used whenever a segmented coaxial detector is needed. To obtain reasonably accurate coaxial detector segmentation, the outer detector contact must be the segmented contact. The most conveniently segmented conventional outer contact is the boron-implanted outer contact of an n-type coaxial detector. The ability to segment the outer boron contact is the reason segmented n-type coaxial detectors are suggested for use in low background gamma-ray measurements. However, n-type coaxial detectors should only be used in environments where radiation damage is a concern and/or a thin outer detector

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contact is desired. Segmented p-type (conventional electrode) coaxial detectors would have technical and financial advantages in low background counting experiments.

P-type coaxial detectors are significantly less expensive and have better gamma-ray energy resolution than n-type coaxial detectors. Fundamentally, this is due to the presence of electron-trapping sites found in even the best detector-quality germanium. A small percentage of the electrons arising from gamma-ray interactions in the detector are trapped before reaching the electron-collecting contact. The charge is trapped for a sufficient duration and is not included in the processed signal for that event. The resulting pulse-height deficits cause broadening of gamma-ray peaks. The magnitude of this energy-resolution degradation from electron trapping is strongly dependent on the geometry of the detector. In detectors of coaxial geometry, the charge carriers collected on the inner contact are responsible for inducing most of the total signal from gamma-ray interactions occurring in most of the volume of the detector. In n-type coaxial detectors, electrons are collected on the inner contact. Consequently, the gamma-ray energy resolution of n-type coaxial detectors is degraded by even small amounts of electron trapping. On the other hand, the spectroscopy of p-type detectors of coaxial geometry relies more heavily on the collection of holes on the inner contact. As a result, electron trapping causes much less resolution degradation in a p-type coaxial detector than in an n-type coaxial detector. The decreased sensitivity to electron trapping makes a greater fraction of the germanium crystals viable for fabrication into p-type coaxial detectors having excellent energy resolution. The lesser importance of electron trapping allows fabrication of larger diameter p-type coaxial detectors. Thus fewer detectors are needed to make an array having a given total volume. It is important to note that electron trapping is still not thoroughly understood and difficult to control in the growth of detector-quality germanium. Any large-scale low-level counting facility employing segmented coaxial detectors would greatly benefit, both technically and financially, from the use of p-type coaxial detectors.

Currently the segmentation of the outer lithium-diffused n+ contact of a p-type coaxial detector is a nontrivial operation. The outer contact of a p-type coaxial detector is conventionally made using a rather thick (as much as ~1 mm thick) lithium-diffused layer as the hole-barrier contact. Thick lithium-diffused contacts are very rugged and reliable but require rather drastic techniques for segmentation. Some techniques involve cutting through the lithium-diffused layer with a saw to segment the contact. Although it can work, such detector fabrication techniques are expensive, time consuming, and mechanically cumbersome. In the event that a saw-cut lithium contact does not successfully function, successive fabrication attempts may prove difficult. Accommodating the saw-cut grooves during the subsequent fabrication attempts may be sufficiently complicated to compel regrinding the crystal to a smaller diameter or even starting over again with a new crystal. In addition, such saw cuts can cause charge-collection and surface-channel problems in the vicinity of the grooves between the segments. Grooves often result in effectively “dead” germanium near the grooves. The initial saw cuts and electronically “dead” germanium consume valuable isotopically enriched germanium.

To make segmented p-type coaxial detectors viable, better outer contacts must be developed to replace saw-cut segmented thick lithium contacts. There are other contact technologies with the potential to provide hole-barrier contacts that are more easily segmented than thick-lithium n+ (hole barrier) contacts. This study seeks to determine the best solution for producing thin-segmented hole barrier contacts on p-type germanium detectors. This will make p-type coaxial detectors viable for large-scale low-level counting arrays. We have started investigating alternative techniques for making segmented hole-barrier contacts in lieu of conventional thick lithium-diffused n+ contacts. Amorphous germanium contacts represent one possible alternative. During Phase I of this SBIR we fabricated and tested many small planar test detectors (~2–4 mm thick, ~30-mm diameter) having segmented amorphous germanium contacts as the hole-barrier (+ biased) contacts. We focused on making the amorphous germanium hole barrier as high as possible. A larger hole barrier provides better rectification and a higher probability of successful fabrication of large diameter p-type coaxial detectors using the thin amorphous germanium contact over the entire outside area of the detector. Amorphous germanium contact technology naturally lends itself to the simple fabrication of finely segmented germanium detectors (Luke et al., 1992; Hull et al., 2002; and Hull et al., 2003). By making many planar test detectors, we studied the rectification and segmentation of amorphous germanium contacts with a focus on increasing the hole-barrier contact. We demonstrated the viability of our fabrication techniques by fabricating a small p-type coaxial detector (MJ1) having an amorphous germanium outer contact. The successful rectification of the amorphous germanium contact over the large curved outer contact of MJ1 serves as a first step toward a viable manufacturing process for segmented p-type coaxial detectors. This work was sufficient to provide a successful Phase 2 application (Hull, E. 2005, Hull, E. 2006).

During the first year of Phase 2, significant strides have been made to analyze the potential for the fabrication of segmented lithium-diffused contacts on germanium detectors. In principle, lithium-diffused contacts could be used to form rectifying hole barriers that are segmented. The goal of Phase 2 is the research of segmented lithium contacts by fabrication of planar and coaxial germanium detectors.

RESEARCH ACCOMPLISHED

Initially, effort was dedicated to building a lithium evaporation and diffusion station for evaluating the limitations of segmented lithium-diffused contacts on germanium detectors. A Veeco 7760 thermal vacuum evaporation system was converted into a system suitable for evaporation and thermal diffusion of lithium into germanium. A vacuum housing and shielding structure was designed and built. These shields prevent the outer vacuum vessel and vacuum sealing surfaces from becoming coated with lithium during the evaporation process. A manually actuated shutter was installed to control the duration of the thermal evaporation of lithium from the source onto the detectors. Figure 1 is a photograph of the lithiation station when closed. A current supply capable of producing .75 Amps at 100 V is used to drive current in a Zener diode on a heating plate in the vacuum system. The detectors are heated on this plate to diffuse the evaporated lithium into the germanium. The temperature of the plate was read out using a DT670 LakeShore diode. The temperature measurement requires reading the voltage drop on the diode when 10 microamps of current is supplied to the diode.

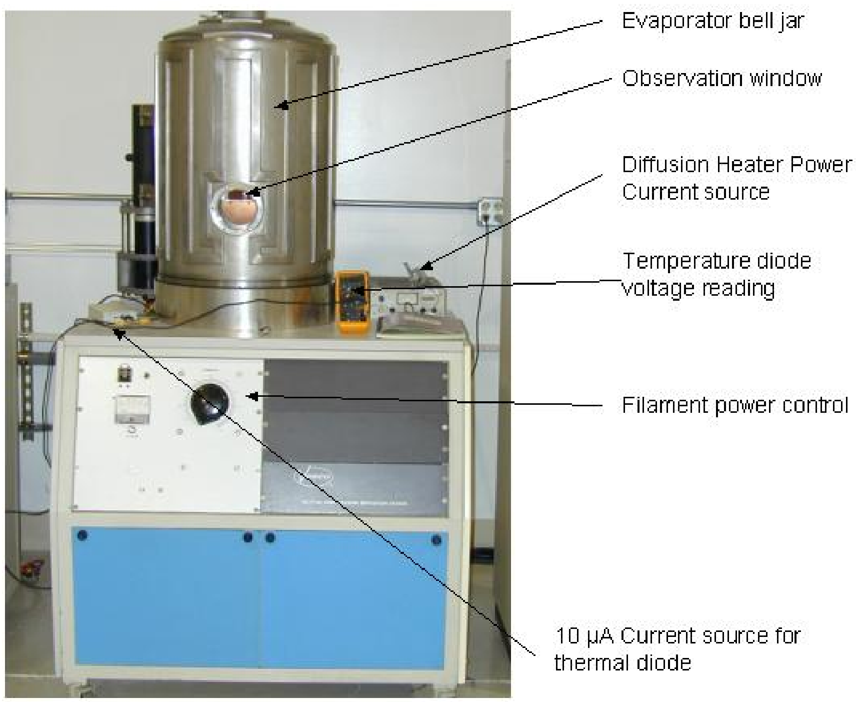

Figure 1. The lithiation system provides a high vacuum environment for the evaporation and diffusion of lithium into germanium.

A procedure for low temperature lithium diffusion was developed using small planar test detectors. The detectors were etched in a mixture of 3:1 HNO_3:HF and rinsed in methanol. The temperature of the plate was increased after the lithium was evaporated. The detectors were warmed to ~ 100º C for ~ 1hr. The detectors were then cooled to ~ 80º K in a vacuum test cryostat. The rectification and separation of the lithium contacts were tested the following morning. These detectors showed some signs of rectification, but the rectification was so poor that most of the

detectors would only withstand an electric field of ~ 200 V/cm before the diode broke down. This is not nearly enough electric field to operate a large coaxial detector. Even at electric fields less than breakdown, these detectors showed very poor looking gamma-ray signals characteristic of a non-rectifying semiconductor junction. These detectors also exhibited poor segmentation between the center and guard-ring contacts of the detector.

The temperature of the lithium diffusion was increased to ~125° C for ~ 1 hr, by sending more power to the Zener diode in the diffusion hot plate. This relatively small increase in temperature improved the rectification of the lithium contact tremendously. After the ~125º C diffusion, the detectors rectified well enough to tolerate electric fields of ~ 1000-4000 V/cm and showed much more reasonable looking gamma-ray signals when the center and guard ring segments were shorted together externally and sent to a single preamplifier. The signals were still very noisy, typically $FWHM_{noise}$ = 3-5 keV. In addition, the center and guard-ring segments of the detector were not separating well. That is why the leads connected to the segments were shorted together and sent to a single preamplifier. The first few detectors diffused at 125°C had an inter-electrode resistance of ~ 200 Ω between the center and guard-ring segments. With the preamplifiers typically having feedback resistor values of at least 1 GΩ and operating voltages of a ~ 0.1 V, there is no possibility to form a segmented germanium detector with such a low inter-electrode impendence.

To address the problem of segmentation, a brief (1 second) etch was attempted on the segmented surfaces of four of the working detectors with 3:1 HNO_3:HF. This completely ruined the rectification of all but one of the four detectors. The single functioning detector remained segmented with an inter-electrode impedance of ~ 200 Ω. Performing a following etch also has the disadvantage of making the segmentation lines on the detector completely disappear. The segmentation pattern left by the lithium shadow-mask evaporation is very faint immediately after the lithium diffusion. However, after a following 1-second 3:1 etch, the lithium-diffusion segmentation pattern is almost completely invisible. This is an extreme disadvantage when aligning contact pins for connection to a segmented detector.

More detectors were made using the lithium shadow evaporation technique. The masks were made wider and placed closer to the surface of the germanium during the evaporation. It was hypothesized that the lithium was somehow undershooting the shadow mask; although there was no visible evidence to support this explanation. Unfortunately, the same result was observed regardless of the shadow geometry. The lithium contacts would rectify well but would not segment beyond a few hundred ohms; at least 6 orders of magnitude too low. After many attempts, we became worried that stable segmentation was not possible with closely spaced lithium contacts. Several different shadow evaporation techniques were attempted using different mask configurations.

After several more attempts, a masking technique was finally identified that produced very high impedance between the detector segments. Using this "new technique," inter electrode impedances of ~ 100 GΩ are routinely obtainable while the excellent rectification is maintained. The successful segmentation and rectification of lithium was dramatic and important for this project. In addition, this processing technique leaves a pattern that is also clearly visible on the surface of the detector. This is necessary for the positive alignment of connection pins to the electrodes on multi-segment detectors.

The photograph presented in Figure 2 shows one of the segmented-lithium planar detectors. The segmentation line between the center and guard ring is clearly visible on this 1-mm thick detector. The detector rectified, separated, and operated successfully up to ~ 400 V, providing an electric field of ~ 4000 V/cm.

Figure 2. A 1-mm thick planar test detector has a segmented lithium-diffused contact visible on the top surface. The contact withstood ~ 4000 V/cm electric field and segmented with several hundred GΩ.

To measure the degree of segmentation, the effective resistance between the center and guard-ring electrodes was measured. When the resistance is extremely high, a higher voltage resistance measurement technique must be used. The center segment is allowed to operate normally with a preamplifier having a 1 GΩ feedback resistor. Because of the 1 GΩ feedback resistor, the DC feedback voltage of this preamplifier will change in voltage by 1 mV when a current of 1 pA flows into (or out of) the gate of the JFET. The guard-ring electrode of the detector was then attached to a ~ 10 V DC voltage source to drive current between the guard ring and center contact. A voltage of ~ 10 V is enough to cause a swing in the DC level of the center contact preamplifier but not high enough to significantly affect the electric field inside the detector. This detector was biased at 400 V for these measurements. Using this measurement technique, the detectors made with the new segmentation technique showed an inter-electrode resistance of
~ 50×10^{12} Ω or 50 TΩ. This is a huge resistance. A resistance of several GΩ s is quite sufficient for detector operation. Achieving such a high resistance value means that the gap between the electrodes is fully depleted and there is little or no surface conductivity from surface channels. The 125°C 1-hr lithium diffusion should create a contact that is ~ 100 µm thick and wide. Because our gap is ~ 1 mm, there should be ~ .8 mm of depleted material in the gap region.

After more trials and work on the technique, more encouraging results were obtained. The detectors generally held ~ 3000 V/cm of electric field with no measurable leakage current, and had an interelectrode resistance of ~ 15 TΩ. These detectors were cycled to higher temperatures in the test cryostat to note any change in the interelectrode resistance. The detectors were cycled to 85°C for 5 hrs while the cryostat was pumped with a turbo molecular pump. After this temperature cycle, the detectors still had an interelectrode resistance of ~ 15 TΩ. A subsequent 2-hour temperature cycle to 100°C lowered the measured resistance to ~ 2.5 TΩ, still very high. The most dramatic change observed as a function of these temperature cycles was the point at which the voltage between the center and guard-ring electrodes was sufficient to break down the resistance and create a very large conductive channel current. When the detectors were originally fabricated, the gap could withstand 30 V before breaking down with the detector biased

at 300 V. After the 85° C cycle it took only 22 V and then after the 100° C cycle it took only 15 V to break down the gap. Normally, the only potential difference between the electrodes on a segmented germanium is ~ 0.1 V from the slight differences in the JFETs on the front end of each preamplifier.

CONCLUSIONS AND RECOMMENDATIONS

Segmented lithium-diffused contacts appear quite stable. These contacts should provide a viable segmented hole barrier contact on p-type coaxial germanium detectors for low-level counting and the search for neutrinoless double beta decay.

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